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CORROSION MEETING
VOLUME II

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Cleveland, Onio

LEWIS RESEARCH CENTER
CLEVELAND, OHIO
OCTOBER 2-3
1963



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PROCEEDINGS OF THE

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CORROSION MEETING VOLUME II

Lewis Research Center, Cleveland, Ohio October 2-3, 1963

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Socrates Christopher, Chairman

J. H. DeVan, A. Taboada, and W.C. Thurber
Oak Ridge National Laboratory
Oak Ridge, Tennessee

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OAK RIDGE NATIONAL LABORATORY

J. H. DeVan, A. Taboada, and W. C. Thurber

I. REFLUXING POTASSIUM CAPSULES

- 1. <u>Purpose of Test</u>: To provide quantitative corrosion data on high-strength refractory-metal alloys in contact with refluxing potassium at temperatures from 1100 to 1300°C. These studies are part of the High-Temperature Materials Program at ORNL, sponsored by the AEC.
- 2. Fluid: Potassium
- 3. Type of Test: Refluxing capsule material to be evaluated is in contact with both liquid potassium and potassium vapor.
- 4. Materials: The following materials will be tested:

Alloy	Starting Form	Surface Conditions	Heat Treatment
Nb-1% Zr	Tubing	Machined, degreased in HNO ₃ + HF	l hr, 1300°C in vacuum
Nb-10% W-1% Zr	Tubing	Machined	2 hr, 1600°C in vacuum
Mo-0.5% Ti-0.08% Zr	Tubing	Machined	Undecided
Ta-8% W-2% Hf	Tubing	Machined	Undecided

- 5. <u>Test Specimens</u>: Tight-fitting, sleeve-type inserts lining the inside diameter of the upper or condenser section of the capsule. The capsule, specimens, and end plugs will be made of the same material.
- 6. Test Configuration: A schematic of the test system is shown in Fig. I-1.

7. Control Variables Studied:

		Tes	st Variables	
Alloy	Temperature (°C)	Time (hr)	Oxygen Concentration in Potassium (ppm)	Refluxing Rate (g/min)
Nb-1% Zr	1100	5000	< 100, 500	25–35
	1200	5000	< 100	Unknown
Nb-10% W-1% Zr	1200	5000	< 100, 500	Unknown
	1300	5000	< 100	Unknown
Mo-0.5% Ti-0.08% Zr	1200	5000	< 100	Unknown
Ta-8% W-2% Hf	1200	5000	< 100, 500	Unknown
	1300	5000	< 100	Unknown



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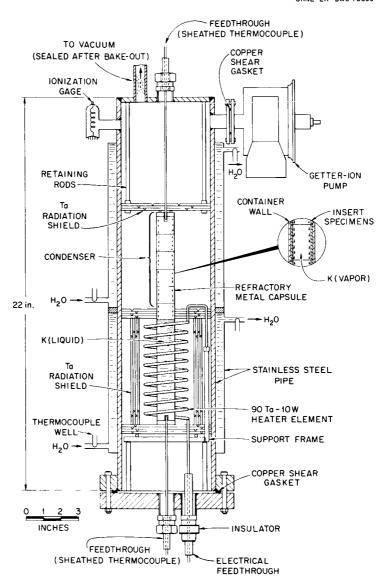


Fig. I-1. Refractory Metal-Refluxing Potassium Capsule Test.





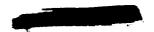
- 8. Purity of Alkali Metal: The potassium to be used in these studies was obtained from the Mine Safety Appliance Company. Duplicate prepurification analyses for oxygen concentration by the mercury amalgamation procedure were 43 and 52 ppm. Potassium was then purified by heat treating for 24 hr at 760°C while in contact with zirconium foil. Duplicate post-purification analyses for oxygen concentration by the mercury amalgamation procedure were 24 and 26 ppm.
- 9. <u>Method of Loading and Sealing Capsules</u>: Capsules are filled with weighed amounts of liquid potassium under argon in an atmosphere chamber. An end plug is then welded on the capsule. The capsule is sealed under vacuum by autoresistance welding the evacuation line from the end plug.
- 10. <u>Test Environment</u>: Vacuum $< 5 \times 10^{-8}$ torr. Pressure is measured by ionization gage and getter-ion pump current.
- 11. Fluid Flow Rate: Potassium condensing rates of 25 to 45 g/min are expected in these systems. Rates will be determined from measurements of the temperature rise and flow rate of water in a jacket surrounding the condenser section of the system.
- 12. <u>Flow Stability</u>: Unknown. Instabilities generally have been observed to be of high frequency and low magnitude at temperatures of 1000°C or higher. Temperature monitoring of condenser and boiler will be the main criterion determining stability of boiling rate.
- 13. <u>Method of Heating and Control</u>: As shown in Fig. I-1, a resistance-heated helix made of 90 Ta-10 W alloy is used to heat the boiler section of the capsule. The power input to the heater is manually controlled through a Variac.
- 14. <u>Instrumentation</u>: Temperature of the system is measured by Pt_{90} Rh_{10}/Pt or W_5 Re/W_{26} Re thermocouples located in wells extending into the boiler and condenser regions (Fig. I-1). An additional Chromel-P/Alumel thermocouple is located on one of the heat reflectors surrounding the boiler section. Temperatures are continuously recorded on a multipoint recorder. Pressure

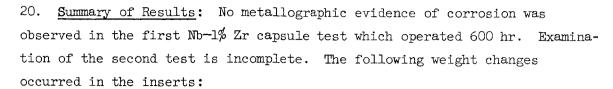




of the system is measured with an ionization gage and control. A getterion pump is used to maintain a vacuum of $< 5 \times 10^{-8}$ torr and can also be used to measure the pressure of the system. Water temperatures are measured by Chromel-P/Alumel thermocouples extending into wells in the inlet and exit lines. Water flow is measured with a graduated flask and a timer.

- 15. <u>Posttest Procedure</u>: Power to heater is turned off to terminate the test. The capsule is removed from the test chamber and visually inspected. The capsule is cut open in inert-gas atmosphere chamber and insert specimens are removed. Samples of potassium are cut for chemical analyses and loaded into glass ampoules.
- 16. <u>Method of Measuring Corrosion</u>: Weight change in insert specimens is the first means of measuring corrosion. Metallographic examination of inserts as well as specimens from the boiler and condenser walls is then made. Chemical analyses are used to determine if changes in composition have occurred in either specimens or capsule. Further examination will depend on the results found using the procedures mentioned above.
- 17. Reproducibility of Results: Unknown. No base-line data yet available nor have there been enough tests on the same system to evaluate reproducibility.
- 18. Number of Premature Failures Due to Causes Other Than Corrosion: Two Nb-1% Zr tests have failed due to rupture of the capsule wall. First failure was attributed to drift of Pt₉₀ Rh₁₀/Pt thermocouples. Power to heater was increased to maintain indicated temperature at 1100°C. True temperature was probably in excess of 1300°C during later stages of test. Rupture occurred after 600 hr. Second Nb-1% Zr capsule ruptured after 3200 hr at 1100°C. No satisfactory explanation for this failure has been found.
- 19. Man Power Involved: One engineer plus one technician.

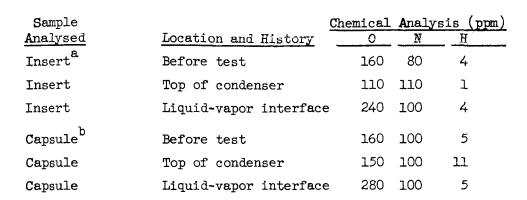




		Weight Cha	nge (mg/cm ²)
Location	Insert No.	Nb-1% Zr 600 hr at 1100-1300°C	Nb-1% Zr 3200 hr at 1100°C
Top of	1	-2×10^{-5}	-10.6×10^{-5}
condenser	2	-4×10^{-5}	-23.7×10^{-5}
	3	-4×10^{-5}	-10.6×10^{-5}
	4	$+1.5 \times 10^{-5}$	+49.1 × 10 ⁻⁵
₩	5	+22 × 10 ⁻⁵	$+80.7 \times 10^{-5}$
Liquid- vapor interface	6	+30 × 10 ⁻⁵	+31.7 × 10 ⁻⁵

The pattern of small weight losses in the upper portion of the condenser with somewhat larger weight gains in the vicinity of the liquid-vapor interface is characteristic of results which have been observed in previous refluxing capsule and natural-circulation boiling-potassium loop tests. Overall weight losses are of small absolute magnitude. The average weight loss for the 600 hr test was approximately 3×10^{-5} mg/cm² which corresponds to the uniform removal of 1×10^{-6} in. from the surface. The average weight loss for the second test was 15×10^{-5} mg/cm² which gives a uniform removal of only 5×10^{-6} in. In both tests the average rate of dissolution was calculated to be approximately 5×10^{-8} mg/cm²-hr.

Results of chemical analyses of inserts and capsule wall from the 600 hr test are given as follows.



^aSpecimen thickness: 0.040 in.

Very little interchange of interstitials between Nb-1% Zr and potassium occurred. It does appear that the Nb-1% Zr which was in contact with liquid potassium did getter a small amount of oxygen from it.

II. NATURAL-CIRCULATION BOILING-POTASSIUM CORROSION LOOP TESTS

A. Type 316 Stainless Steel Loop

1. <u>Purpose of Experiment</u>: To determine the dissolution and deposition rates of the loop material in the condenser and subcooler regions at a known condensing rate. These tests are sponsored by the AEC under the High-Temperature Materials Program at ORNL.

2-4. Description of Test System:

- a. <u>Fluid</u> High-purity, low sodium grade potassium. Filtered, titanium gettered, and cold trapped.
- b. Manner of circulation Boiling, naturally circulating.
- c. Overall size and general design vertical legs: 6 ft horizontal legs: 1.5 ft Composed of boiler, vapor carry-over line, condenser, subcooler, and preheater.

For configuration, see attached schematic drawing (Fig. II-A-1).



bWall thickness: 0.050 in.



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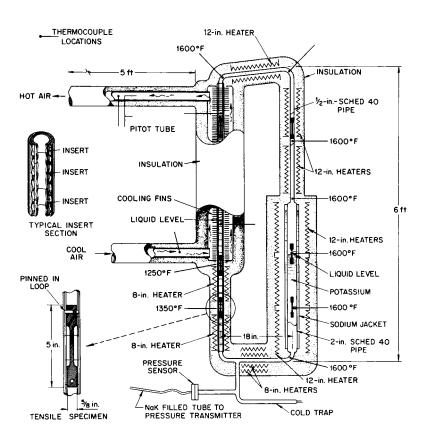


Fig. II-A-1. Boiling-Potassium Loop.





5. Containment Alloys:

- a. Alloy Type 316 stainless steel.
 - 1. Tubing purchased according to ASTM Designation: A 213 60 T.
 - 2. Seamless pipe purchased according to ASTM Designation: A 376 61 T.
- b. Sizes 1/2 in., 3/4 in., and 2 in. pipe, (all sched 40); 1/4-in.-OD by 0.065-in.-wall tubing.
- c. Assembly method Heliarc welding.

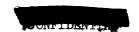
6. Test Conditions:

- a. Temperatures:
 - 1. Boiler 880°C (1620°F)
 - 2. Condenser 870°C (1600°F)
 - 3. Subcooler 675°C (1245°F)
- b. Pressures System pressure cycled from 39 to 46 psia.
- c. Flow Rate 180 g/min as calculated by a heat balance around the condenser.
- d. Operating time 3000 hr.

7. Test Specimens:

- a. <u>Tubular Inserts</u> Forty-four specimens, each 1 1/2 in. long, were machined from 1/2-in. sched-40 pipe, weighed and identified, and stacked in the vertical condenser-subcooler leg. Machining the inserts provided a 5-mil annulus between the inserts and the inside diameter of a 3/4-in. sched-40 cover.
- b. <u>Tensile Specimens</u> Sheet specimens, 0.040-in. thick, were suspended on anchor pins in various loop regions. Specimens were H₂ fired at 1070°C for 30 min.
- c. Removal of Test Specimens All insert specimens were removed at the completion of the test, weighed for weight changes, and examined along with other sections of the loop. Tensile specimens were removed and subjected to standard tension tests.





8. Pretreatment of Loop:

- a. All loop components prior to assembly were subjected to standard x-ray and eddy-current inspection techniques. Heliarc welds were inspected according to standard x-ray and dye-penetrant inspection.
- b. Assembled loop was degreased with acetone and evacuated prior to admitting the potassium charge.
- 9. <u>Method of Filling Loop</u>: Predetermined amount of charge was admitted by inert-gas pressurization.
- 10. <u>Startup Procedure</u>: Apply intermediate power to vapor carry-over lines and preheaters; gradually bring boiler up to temperature; adjust other heaters.
- 11. Test Environment: Air.

12. Instrumentation:

- a. Pressure Taylor gage with NaK-filled sensor tube.
 - 1. Accuracy 0.47% full scale.
 - 2. Operating life unknown.
- b. <u>Temperature</u> Chromel-P/Alumel thermocouples and 10 mv, 12-point recorder.

13. Description of Components:

- a. Heaters Kanthall-wound, direct resistance, "Clamshell-type."
- b. <u>Boiler</u> 36-in. long, 2-in. sched-40 pipe surrounded by a 4-in. sched-40 Inconel sodium jacket.
- c. Condenser Tubular insert-type as described in 7a.
- 14. <u>Alkali-Metal Sampling</u>: Sample tube was connected in series with fill line and sample collected as loop was being filled. During draining, samples were taken in the same manner.

Initial and final samples: approximately 200 ppm oxygen content based on mercury amalgamation method.





15. <u>Loop Flow Stability</u>: Unstable boiling conditions prevailed. System pressure fluctuated between 39 and 46 psia.

16. Posttest Procedure:

- a. Visual examination with fluorescent penetrant (Zyglo) technique.
- b. Extensive metallographic examination.
- c. Chemical analyses of (1) loop material, (2) tubular inserts,(3) tensile specimens, (4) mass transfer deposit, and (5) potassium.
- d. Mechanical tests on tensile specimens.

17. Major Loop Problems:

- a. Unstable boiling.
- b. Thermal fatigue cracks caused by unstable conditions as a result of (a).

18. Man Power:

- a. During design 1 engineer.
- b. During setup -1/2 technician, 2 craft.
- c. During operation -1/2 technician.
- 19. Summary of Results: (Table II-A-1).





Table II-A-1 Type 316 Stainless Steel-Boiling Potassium Natural-Circulation Loop No. 2

Operating Conditions:

Temperatures: Boiler - 870°C; Subcooler - 675°C

Flow Rate: 180 g/min (24 lb/hr)

Vapor Velocity: 50 fps in 1/2-in. sched-40 pipe

Test Duration: 3000 hr

Reason for termination: scheduled

Results:

Weight change of inserts:

Condenser region: -8 mg/in.² (uniform)

Subcooled liquid region: +90 mg/in.² (maximum)

Metallographic examination:

Boiler (liquid - 870°C); surface roughening to 1 to 2 mils in depth

Boiler (liquid-vapor interface - 870°C); surface roughening to 2 mils

Condenser (870°C): subsurface voids to a depth of 10 to 12 mils

Condenser (liquid-vapor interface): severe cracking

Subcooled liquid (675°C): 8 mils mass transfer deposit;

carburization in excess of 10 mils

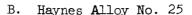
Carbon transfer:

As-received material: 0.10% C

Condenser (9 mil layer): 0.04% C

Subcooler (9 mil layer): 0.41% C





1. <u>Purpose of Experiment:</u> To determine the dissolution and deposition rates of the loop material in the condenser and subcooler regions at a known condensing rate. These tests are sponsored by the AEC under the High-Temperature Materials Program at ORNL.

2-4. Description of Test System:

- a. <u>Fluid</u> High-purity, low sodium grade potassium. Filtered, titanium gettered, and cold trapped.
- b. Manner of Circulation Boiling, naturally circulating.
- c. Overall size and General Design vertical legs: 10 ft
 horizontal legs: 3 ft
 Composed of boiler, vapor carry-over line, condenser, subcooler,
 and preheater (shown in schematic Fig. II-B-1).

5. Containment Alloys:

- a. Alloy Haynes Alloy No. 25
 - 1. Seamless tubing
 - 2. Seamless pipe (except 1/2-in. sched-40)
- b. Sizes -1/2-in., 3/4-in., and 2-in. pipe (all sched-40); 1/4-in.-OD by 0.065-in.-wall tubing.
- c. Assembly Method Heliarc welding.

6. Test Conditions:

- a. Temperatures:
 - 1. Boiler 980°C (1800°F)
 - 2. Condenser 980°C (1800°F)
 - 3. Subcooler 800°C (1470°F)
- b. Pressures System pressure cycled from 45 to 80 psia.
- c. Flow Rate 300 g/min as calculated by a heat balance around the condenser.

7. Test Specimens:

a. <u>Tubular Inserts</u>: Twelve 9-in.-long tubes accurately machined from 3/4-in. sched-40 pipe. A weight per unit length was obtained on these prior to machining bevels for welding. They were then welded together to form the condenser-subcooler leg.





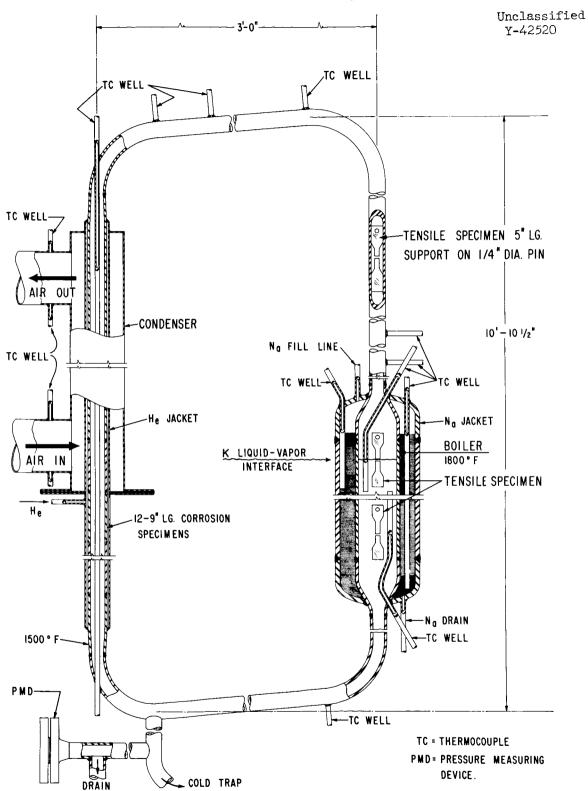


Fig. II-B-1. Natural-Circulation Boiling-Potassium Corrosion Loop.



7. Test Specimens (continued):

- b. <u>Tensile Specimens</u>: Sheet tensile specimens (0.040-in. thick) suspended on welded pins.
- c. Removal of Test Specimens: Tubular insert specimens were removed by (1) cutting condenser-subcooler leg in welds, and (2) machining square ends on tubes. The weight change per unit length was obtained and a weight loss or gain per unit condensing area was calculated.

Tensile specimens were removed and subjected to standard tensile tests at room temperature and in 980°C air.

8. Pretreatment of Loop:

- a. All loop components prior to assembly were subjected to standard x-ray and ultrasonic inspection techniques. Heliarc welds were inspected according to standard x-ray and dye-penetrant inspection.
- b. Assembled loop was degreased with acetone and evacuated prior to admitting the potassium charge.
- 9. <u>Method of Filling Loop</u>: Predetermined amount of charge was admitted by inert-gas pressurization.
- 10. <u>Startup Procedure</u>: Apply intermediate power to vapor carry-over lines and preheaters; gradually bring boiler up to temperature; adjust other heaters.
- 11. Test Environment: Air.

12. Instrumentation:

- a. Pressure Taylor gages with NaK-filled sensor tubes.
 - 1. Accuracy 0.47% full scale.
 - 2. Operating life unknown.
- b. <u>Temperature</u> Chromel-P/Alumel thermocouples and 10 mv, 12-point recorder.



13. Description of Components:

- a. <u>Heaters</u> Kanthall-wound, direct resistance, "Clamshell-type."
- b. <u>Boiler</u> 36-in. long, 2-in. sched-40 pipe surrounded by a Haynes Alloy No. 25 sodium jacket fabricated from 1/4-in. plate.
- c. Condenser Tubular insert-type as described in 7a.
- 14. <u>Alkali-Metal Sampling</u>: Sample tube was connected in series with fill line and sample collected as loop was being filled. During draining, samples were taken in the same manner.

Initial and final samples: 20 ppm and 50 ppm oxygen content, respectively, as determined by the mercury amalgamation procedure.

15. Loop Flow Stability: Unstable boiling conditions prevailed. System pressure fluctuated between 45 and 80 psia. These cycles were accompanied by loop temperature cycles of approximately 10°C. Frequency of cycles varied from approximately 20 sec to 1 hr.

16. Posttest Procedure:

- a. Visual examination with fluorescent penetrant (Zyglo) technique.
- b. Extensive metallographic examination.
- c. Chemical analyses of (1) loop material, (2) tubular inserts,(3) tensile specimens, (4) mass transfer deposit, and (5) potassium.
- d. Mechanical tests on tensile specimens.

17. Major Loop Problems:

- a. Unstable boiling.
- b. Thermal fatigue cracks as a result of unstable conditions.

18. Man Power:

- a. During design 1 engineer.
- b. During operation -1/2 technician.

19. Summary of Results:

- a. Weight changes
 - 1. Tubular inserts: weight losses in condenser, 1.6 to 2.8 mg/cm² (10 to 18 mg/in.²); weight gains in subcooler, 6.4 to 10.8 mg/cm² (40 to 70 mg/in.²).



19. Summary of Results (continued):

- b. Metallographic Examinations
 - 1. Boiler no attack detected.
 - 2. Condenser surface roughening to 1/2 mil in depth.
 - 3. Subcooler cracks to a maximum depth of 15 mils. Mass transfer deposit to a maximum thickness of 10 mils.
- c. Tensile tests (see Table II-C-1).

Table II-C-1 Results of Tensile Tests of Haynes Alloy No. 25 Specimens from Boiling-Potassium Loop Operated for 3000 hr at a Boiler Temperature of 980°C

Temperature of Tensile Test (°C)	Specimen a Exposure Environment	Elongation in 2 in. (%)	Tensile Strength (psi)	Yield Strength ^b (psi)
Test specimens from loop				
Room	Boiler liquid	19.5	130.3	56.2
980	Boiler liquid	50.0	18.5	15.9
Room	Boiler liquid-vapor interface	18.5	137.7	60.0
980	Boiler liquid-vapor interface	57.5	20.5	16.2
Control specimens				
Room 980	Heat treated in vacuum Heat treated in vacuum	23.5 48.5	153.0 22.3	66.9 18.1

^aAll specimens were 0.040-in. thick, annealed at $2150^{\circ}F$ ($1180^{\circ}C$) for 30 min and water quenched prior to exposure; tests conducted in air.

bAt 0.2% offset.

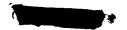


C. Refractory Metals

1. <u>Purpose of Experiments</u>: To study the corrosion and mass transfer effects produced by nonisothermal boiling potassium in refractory-alloy systems. The experiments are conducted under the AEC-sponsored High-Temperature Materials Program at ORNL.

2-4. <u>Description of Test System:</u>

- a. <u>Fluid</u> 99.9% potassium (liquid plus vapor). Filtered at 200°C and hot gettered with titanium sponge for 250 hr at 650°C and 24 hr at 760°C.
- b. Manner of Circulation Natural convection of vapor between boiler and condenser sections.
- c. Loop Design Rectangular profile composed of two 30-in. vertical sections connected by two 14-in. horizontal sections. Preheater, boiler, and condensing sections constructed of approximately 1-in.-OD by 0.050-in.-wall tubing. Vapor carry-over line constructed of 0.4-in.-OD by 0.040-in.-wall tubing. (See attached schematic drawing, Fig. II-C-1).
- 5. Containment Alloys: The refractory alloys selected for testing under the subject program include Nb-1% Zr, Nb-10% W-1% Zr (X-110), and Ta-8% W-2% Hf (T-111). The latter two alloys are being supplied through AEC-NASA-Air Force tubing-development contracts. All alloys will be tested in the form of seamless tubing supplied to dimensions and specifications listed in Table II-C-1. Loops will be assembled by tungsten arc-inert gas welding and welds will be heat treated to establish a "stabilized" microstructure.
- 6. Test conditions of refractory-metal-boiling potassium, natural-circulating loops (see Table II-C-2).
- 7. Test Specimens: Machined inserts, depicted in Fig. II-C-1, are incorporated in the condenser and subcooler sections to permit weight change determinations. The inserts are removed at the end of the scheduled test period, weighed, and examined metallographically. Several sections of loop piping are also removed for metallographic and chemical studies after test termination.



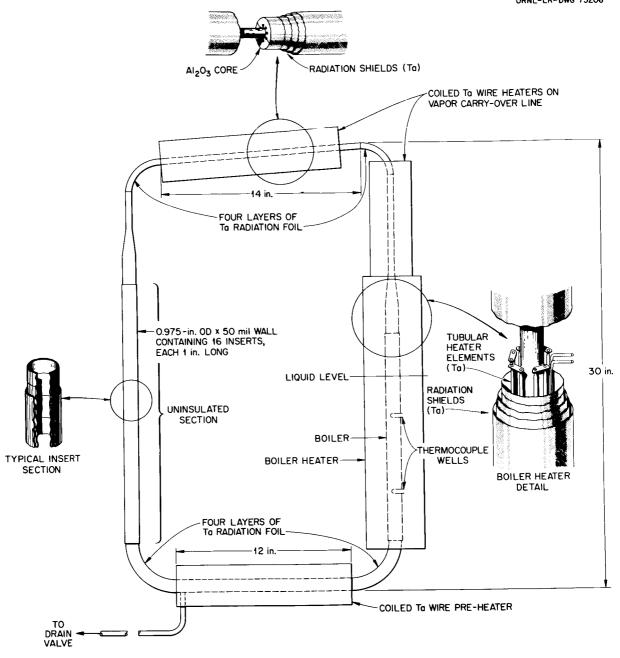


Fig. II-C-1. Refractory-Metal Natural-Circulation Boiling-Potassium Loop.



Table II-C-l Alloy Compositions and Sizes Employed in Refractory-Metal-Boiling-Potassium Natural-Circulation Loops

		Sizes of Loop	Tubing		
Alloy	Corrosion Inserts	Condenser, E and Prehea	•	_	or Carry- er Line
Nb-1% Zr ^a	0.86-inOD × 0.040-in. wall	1.00-inOI 0.065-in.			nOD × O-in. wall
Nb-10% V-1% Zr ⁸ (X-110)	0.370-inOD × 0.057-in. wall	0.50-inOD 0.062-in.		• -	-inOD × 2-in. wall
Ta-8% W-2% Hf (T-111)	0.370-inOD × 0.055-in. wall	0.50-inOI 0.060-in.	-		-inOD × O-in. wall
^a Interstit	ial content (max):	Alloy Nb-1% Zr X-110	0 ₂ 100 ppm 625 ppm	N ₂ 100 ppm 34 ppm	C 300 ppm 1190 ppm

Table II-C-2 Test Conditions of Refractory-Metal-Boiling-Potassium Natural-Circulation Loops

		Test No.		
Conditions	1	22	3	4
Alloy composition	Nb-1% Zr	Nb—1% Zr	Nb-10% W-1% Zr	Ta-8% W-2% Hf
Maximum liquid- vapor temperature	2070°F(1130°C)	2200°F(1200°C)	2200°F(1200°C)	2200°F(1200°C
Minimum liquid temperature	1200°F(650°C)	1260°F(680°C)	1400°F(760°C)	1400°F(760°C)
Condensing rate	40 g/min	36 g/min		
Operating time	2800 hr	3000 hr	3000 hr	3000 hr
Test status	completed	in progress	startup approximately January 1964	startup approximately June 1964



- 8. Pretreatment of Loop: Each loop system is acid cleaned prior to welding and cleaned with acetone, followed by alcohol, immediately prior to filling. The loop is then degassed for 6 hr at 150°C and maintained under vacuum until filling with potassium.
- 9. Method of Filling Loop: Potassium at 150°C is transferred to the loop by inert-gas pressurization. After filling, the loop and its contents are degassed under vacuum at 150°C for 1 hr. The potassium is then allowed to freeze and the system sealed under a vacuum of < 10⁻⁵ torr.
- 10. Startup Procedure: The loop together with auxiliary instrumentation is set up within a stainless steel vacuum chamber. After installation, the chamber is sealed and baked out 170°C under a vacuum of 10⁻⁵ torr for 20 hr. The loop is then brought to operating conditions while maintaining a vacuum of < 10⁻⁵ torr.
- 11. Test Environment: Following a startup period of approximately 200 hr, the test chamber attains and holds a base pressure ranging from 4×10^{-7} to 2×10^{-7} torr. During the startup period, which is used to bring the loop to design conditions, outgassing rates are controlled to hold the system vacuum below 10^{-5} torr. Pressures are measured by ion gages attached to the chamber and vac-ion pump. A residual gas analyser is used to record gas content. Typical analyses obtained throughout loop operation are shown in Table II-C-3.

Additional protection against contamination by residual impurities is afforded by tantalum wrapping wound tightly over the vapor carry-over and preheater sections.

12. Instrumentation:

- a. <u>Temperature measurement</u>: Boiler, condenser, and subcooler temperatures are recorded using W_5 Re/ W_{26} Re thermocouples positioned in wells inserted radially into the loop piping. An additional measure of the condenser temperature is provided by a viewing port and PYRO-EYE two-color optical pyrometer system.
- b. Residual gas analyses Type 21-612, manufactured by Consolidated Electrodynamics Corporation.





Table II-C-3 Analysis of Test Environment During Operation of Nb-1% Zr-Boiling Potassium Loop No. 1

		Partial	Partial Pressure (torr	corr)		Total Pressure (torr) ^a
		四)	(mass/charge)			(Ion Gage,
Condition of Chamber	2 (H ₂)	18 (H2O)	28 (N2,CO	28 (N2,CO) 32 (O2)	40 (Ar)	Corrected)
Cold, initially	1 × 10-6	9 × 10-7	1	4 × 10-7	4 × 10-7	7.5 × 10 ⁻⁶
Bake out	1×10^{-5}				1 × 10-6	4 × 10 ⁻⁵
Loop heatup	4 × 10 ⁻⁵	2 × 10 ⁻⁵	3 × 10.5	5×10^{-7}	1 × 10-6	1 × 10-4
Loop operating						
0 hr	2×10^{-6}			7 × 10-8	6×10^{-7}	7 × 10 6
100 hr	2×10^{-6}	2×10^{-7}	1 × 10-6	2 × 10-8	3×10^{-7}	4 × 10 ⁻⁶
1000 hr	1 × 10-7		1 × 10-6	2×10^{-8}	2×10^{-7}	1.5 × 10 ⁻⁶
1500 hr	9 × 10-9	8 × 10-8	1 × 10-7	$<1\times10^{-9}$	5 × 10-8	4 × 10 ⁻⁷

 $^{\mathrm{a}}\mathrm{Sensitivity}$ of gage for various gases used to correct indicated pressure.

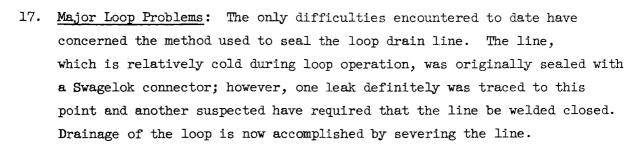
13. Description of Components:

- a. <u>Heaters</u>: The main boiler heating elements are fabricated from 3/16-in.-diam \times 0.015-in.-wall flattened tantalum tubes. Twelve such elements are welded in series in a serpentine configuration which is suspended on Al_2O_3 insulators attached to a 1/16-in.-thick tantalum cylindrical shell.
- b. Condenser Heat Exchanger: Heat is extracted from the condenser by use of a heat exchanger containing a water-cooled coil. Dowtherm is used as a heat transfer media between the inside jacket of the heat exchanger (facing the loop condenser) and the water coil. A heat balance is periodically made on the coolant water in order to establish potassium condensing rate.
- 14. <u>Alkali-Metal Sampling Procedure</u>: Samples of potassium are taken during filling and draining the loop by placing a sample collection tube in series with the fill-drain line.
- 15. Loop Flow Stability: During startup of the initial Nb-1% Zr loop, instabilities (i.e., large temperature fluctuations) prevailed until the condenser temperature exceeded 1000°C. Above this temperature, instabilities completely disappeared. Succeeding loops have incorporated hot fingers in the boiler section, which have effectively prevented instabilities even at low condenser temperatures.

16. Posttest Procedures:

- a. Loop drained under protective argon atmosphere and potassium samples collected.
- b. Corrosion inserts removed, weighed, and examined metallographically.
- c. Sections cut from loop piping for metallographic and chemical examinations.
- d. Successive 10-mil-thick turnings taken from various loop sections and analyzed chemically for O_2 , N_2 , C, and K.
- e. Tensile specimens machined from loop sections and pulled at room temperature.
- f. Bend tests conducted on sections of loop piping.





18. Man Power

- a. During design 1 engineer.
- b. During fitup 1 technician.
- c. During startup -1/2 technician, 1/2 engineer.
- d. During operation part-time technician.
- 19. Summary of Results to Date: A Nb-1% Zr loop was successfully operated for 2800 hr under conditions summarized in Table II-C-4. A potassium condensing rate of 40 g/min and a condenser temperature of 2070°F (1130°C) were maintained. Metallographic examinations and weight change determinations indicated exceptional resistance of the Nb-1% Zr to attack by potassium. Assuming uniform removal of material, maximum weight losses were equivalent to a removal of 6 μin. of the condenser wall. Slight contamination of the Nb-1% Zr alloy was produced by the chamber environment, although contamination was restricted to the outermost regions of the loop piping, and no evidence of embrittlement was detected in mechanical property evaluations of the loop.

A second Nb-1% Zr loop test was less successful and was terminated after 250 hr when traces of potassium were detected on the test chamber walls. Exhaustive leak detection failed to uncover the origin of the leak and the test was replaced by a third Nb-1% Zr loop. As of September 16, 1963, this loop had operated for 840 hr at a condenser temperature of 2200°F (1200°C) and condensing rate of 36 g/min. Evidence of a potassium leak developed early in the operation of this loop in much the same manner as the preceding test. In this case, however, obeying a first impulse, the drain line of the loop, which had been sealed by a Swagelok connector, was clamped and the loop restarted. This expedient proved providential and no further difficulties have been encountered.





Table II-C-4 Test Conditions of Nb-1% Zr Loop Operated with 1130°C Boiler Temperature

General Conditions: Time - 2800 hr

Condenser Temperature - 2000°F (1093°C)

Minimum Subcooled Liquid Temperature-1200°F (650°C)

Average Pressure in Test Chamber -2.3×10^{-6}

Power Input:

Boiler Heater - 2100 w

Vapor Line Heaters - 1300 w

Preheater Return Line - 600 w

Power Input - 4000 w

Power Extracted^a

(by radiation

only)

From Condenser - 3610 Btu/hr (1060 w)

From Subcooler - 805 Btu/hr (235 w)

Power Radiated - 1295 w

Condensing Rate:

35 g/min; 6×10^6 g or 13,000 lb in 2800 hr

0.3 $g/min-cm^2$ (1.9 $g/min-in.^2$)

Vapor Velocity:

1.0 fps in 0.4-in.-diam tube

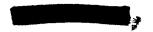
Liquid Velocity:

0.6 fps in 0.8-in.-diam tube

Emissivity = 0.3.

Chamber = a black body.

^aAssumptions: Form factor = 1.



III. FORCED-CONVECTION BOILING-POTASSIUM LOOPS

- A. Type 316 Stainless Steel Loops Containing Various Nozzle and Turbine Blade Materials
- 1. <u>Purpose of Experiments</u>: To conduct screening tests on potential turbine nozzle and blade materials in high-velocity potassium vapor of varying quality and temperature. The experiments are conducted under the AEC-sponsored High-Temperature Materials Program at ORNL.

2-4. Description of Test System:

- a. <u>Fluid</u>: 99.9% purity potassium, filtered at 200°C and hot gettered with titanium sponge at 650°C for 250 hr.
- b. <u>Manner of Circulation</u>: Forced-convection from condenser to boiler using electrodynamic pump.
- c. Loop Design: Trapizoidal profile composed of one 9-ft leg containing 2-in.-diam preheater section, a 4-in.-diam boiler section, a 2-in.-diam drying section, and a second 12-ft leg containing the test section, 2-in.-diam condenser, and subcooler. The two legs are connected by a 4.5-ft vapor carry-over line of 2-in. diam; (see attached schematic drawing, Fig. III-A-1).
- 5. <u>Containment Alloys</u>: The loop, except for nozzle and blade test specimens, is constructed of type 316 stainless steel, sched-40 seamless pipe.

 Assembly of the loop is entirely by Heliarc welding.

6. <u>Test Conditions</u>:

- a. Maximum Boiler Temperature 1600°F (871°C).
- b. Minimum Subcooler Temperature 1000°F (538°C).
- c. Test Section Parameters (see Table III-A-1).
- d. Scheduled Test Duration 3000 hr.
- 7. Test Specimens: The test section consists of a series of three nozzles, each of which discharges its vapor jet against an inclined blade specimen. Nozzles and blades are welded in place, and the entire section must be cut out to be removed. Candidate nozzle and blading materials have, to date, included type 316 stainless steel, Haynes Alloy No. 25, and the Mo-0.5% Ti-0.08% Zr (TZM) alloy.



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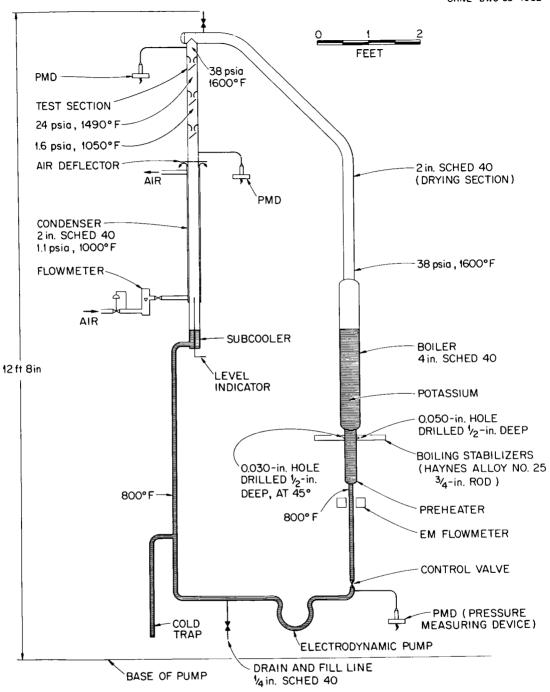


Fig. III-A-1. Type 316 Stainless Steel-Boiling Potassium Nozzle and Turbine Blade Corrosion Loop.

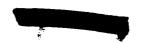
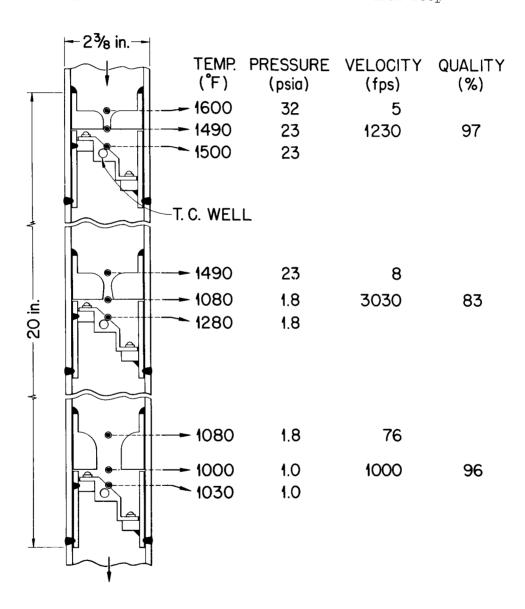


Table III-A-1 Test Section Conditions of Type 316 Stainless Steel-Natural Convection Loop





- 8. Pretreatment of Loop Prior to Filling: The loop is given a flushing treatment by filling with potassium and circulating same for 18 hr at approximately 480°C.
- 9. Method of Filling Loop: The loop is evacuated and baked out at approximately 930°F (500°C) for 18 hr and then charged with potassium by inert gas pressurization. Potassium is admitted at 300°F (150°C) until the proper level is indicated on a level indicator located in the bottom of the condenser.
- 10. Startup Procedure: The loop is started under natural-circulation boiling conditions with hot finger temperatures 93°C above boiler temperature. Pump is started when natural convection flow is insufficient to keep level down in the condenser.
- 11. Test Environment: Air.
- 12. <u>Instrumentation</u>: Refer to Fig. III-A-1. Three Taylor Pressure Measuring Devices (PMD's) are used to monitor pressure in the system. One unit is located downstream from the pump and the other two are at the entrance and exit of the test section. An EM flowmeter is used just below the preheater section while a resistance-level indicator is located in the subcooler. Chromel-P/Alumel thermocouples and multipoint recorders are used to monitor all temperatures.

13. Component Description:

- a. Pump Electrodynamic, manufactured by Liquid Metals, Incorporated.
- b. Heaters Clamshell type composed of Nichrome and Kanthal windings.
- c. <u>Boiler</u> A 36-in.-long 4-in.-sched-40 pipe with an 18-in. long 2-in. sched-40 pipe as preheater.
- d. Condenser A 2-in.-sched-40 pipe, air cooled by counterflow.
- e. <u>Test Section</u> Three nozzles in series, each followed by a blade specimen. The first and third nozzles are converging types while the second is converging-diverging.



- 14. Alkali-Metal Sampling and Analytical Procedure: Samples are taken during filling and draining of loop by forcing a stream through stainless steel tubing thermally equilibrated with the fill pot and system, valving off the stream, and sealing the sample tubes by pinching the inlet and outlets.
 - Analysis for oxygen is done using a modified mercury amalgamation procedure on duplicate samples.
- 15. Loop Flow Stability: Unstable conditions prevailed during the first test of this type (type 316 stainless steel test section). The replacement of the original 1/2-in. sched-40 with a 2-in. sched-40 pipe as the vapor carry-over line failed to prevent slugging of liquid into the test section. Flashing through a control valve aided the instability problem but did not provide conditions that would deliver vapor of the proper quality to the test section.

Completely stable boiling conditions were achieved by employing vapor bubble nucleation sites located on the preheater. These are heated independently of the preheater and their temperature is maintained approximately 93 °C above the temperature in the boiler.

16. Posttest Procedure:

- a. Complete metallographic examination is conducted on all test section and loop components.
- b. Chemical analyses are made on loop components and any mass transfer that can be found.
- c. Complete potassium chemical analyses will be conducted.
- d. X-ray and electron microprobe analyses will be made where applicable.
- 17. Major Loop Problems: Aside from the stability problems described under 15, failures have been experienced in the pump brushes, bearings, and field coils. The resistance level device has been shown to be temperature sensitive. Piping failures, although rare, have occurred. These can usually be traced to a heater element arcing to the pipe wall.

18. Man-Power Review:

- a. Design 1200 man hours.
- b. Fabrication 400 man hours.
- c. Setup 1600 man hours.
- d. Operation 1 engineer, 1 technician.
- 19. Summary of Test Results: The initial loop test to be operated under conditions shown in Table III-A-1 contained a type 316 stainless steel nozzle and blade test section. Extreme boiling instabilities were prevalent throughout this test, and attendant thermal cycling resulted in piping failures after 25 hr and again at 750 hr. The test was terminated after the second failure and the test section and loop walls examined. Only the second-stage blade, which was subjected to the lowest vapor quality and highest vapor quality, showed any detectable operating effects. Impingement damage was noted metallographically on this section to a depth of approximately 1 mil. The impingement area was surrounded by a thin metallic deposit, which was similar in composition to type 316 stainless steel but enriched in manganese.

A second loop test was initiated with TZM nozzle and blade specimens. Design conditions could not be established in this test, however, because of a fabrication error in the test section. This loop is presently being modified, and tests of a second TZM test section will be initiated pending completion of repairs.

A third loop containing a Haynes Alloy No. 25 test section has operated at design conditions for a total of 454 hr. Boiling instabilities were encountered early in the operation of this test, and the test was interrupted to permit installation of a new boiler with provisions for boiling stabilizers (independently heated hot fingers). The loop was restarted and operated very stably until a piping failure occurred. The test will be continued following completion of repairs.



1. Purpose of Experiment: To determine the potential of Nb-1% Zr as a turbine nozzle or blade material for high-velocity potassium vapor of varying quality and temperature. The experiments are conducted as part of the AEC-sponsored High-Temperature Materials Program at ORNL.

2-4. Description of Test System:

a. <u>Fluids</u>: The primary fluid is 99.9% potassium (liquid plus vapor)

The potassium is filtered at 200°C and gettered with titanium sponge immediately prior to use.

A secondary circuit, carrying heat from the condenser, circulates NaK.

- b. Manner of Circulation: Liquid potassium is pumped from the condenser section to the boiler section by an electrodynamic pump. The NaK circuit uses a standard EM pump.
- c. Loop Design: The profile of the loop is roughly rectangular. A 6-ft vertical section of 1.90-in.-OD × 0.125-in.-wall tubing serves as the preheater, boiler, and dryer. There is a sodium jacket on the boiler section. A 1.0-in.-OD × 0.065-in.-wall line is used as a cross-over line between the dryer and the test section. The test section and condenser are constructed of 1.9-OD × 0.125-in. wall tubing. A series of three nozzle and blade specimens are contained in the test section. Condensing heat is removed by conduction to a jacket in which cooling NaK is circulated (refer to Figs. III-B-1 and -2).
- 5. Containment Alloys: The primary loop circuit, sodium jacket, test section (nozzles and blades), and the secondary circuit (condenser) are all constructed of Nb-1% Zr. In the first loop test, the Nb-1% Zr tubing lines going to the electrodynamic pump on the primary circuit and to the electromagnetic pump on the secondary circuit are connected to type 316 stainless steel by tubular transition joints. (See Figs. III-B-1 and -2). All tubing and pipe are seamless; piping dimensions are given in Table III-B-1. The Nb-1% Zr system and test section components are assembled by tungsten arc-inert gas welding in an inert-atmosphere chamber.



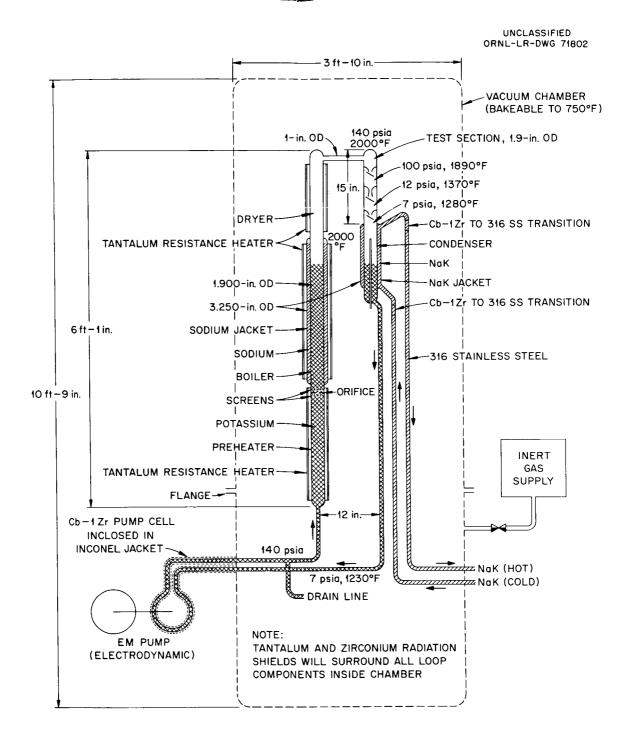


Fig. III-B-1 Nb-1% Zr Boiling-Potassium Nozzle-Turbine Blade Corrosion Loop.



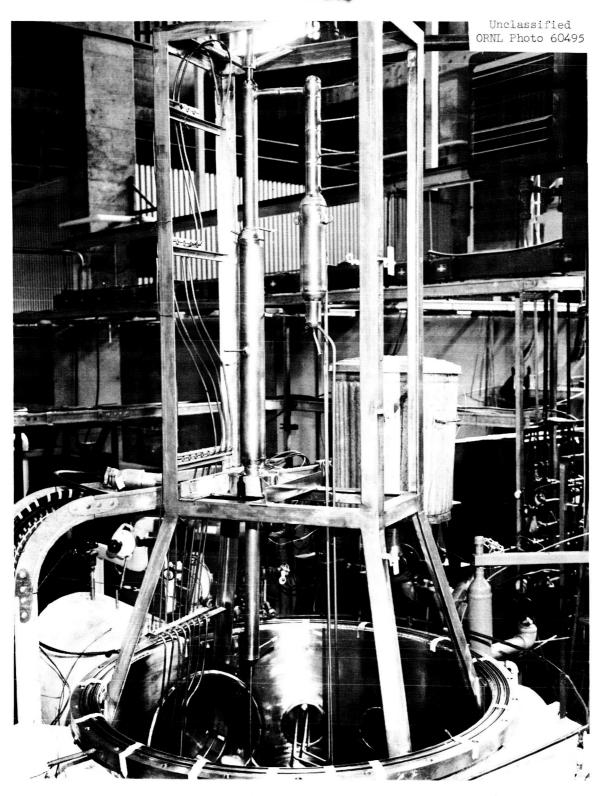


Fig. III-B-2 The Nb-l% Zr Boiling-Potassium Nozzle-Turbine Blade Corrosion Loop and Support Rack as Mounted in the Lower Section of the Vacuum Test Chamber.





Table III-B-1 Vapor Conditions in Nb-1% Zr Forced-Convection Test

Nozzle Location	Vapor Quality (%)	Nozzle Exit Velocity (fps)		ade rature (°C)
First stage	96	1390	1960	1070
Second stage	83	3080	1710	930
Third stage	96	1430	1325	720

6. Test Conditions:

- a. <u>General Conditions</u>: Boiler temperature 1970°F (1077°C)

 Pressure of system 140 psia (saturated vapor)

 Flow rate 32.7 lb/hr

 Time 3000 hr (scheduled)
- b. <u>Test Section</u>: The test conditions at each of the three nozzle blade stations are summarized in Table III-B-1.
- 7. <u>Test Specimens</u>: The test section consists of a series of three Nb-1% Zr nozzle and blade stages. The nozzles and blades are tungsten arc-inert gas welded in place in the test section; the test section is cut out to remove them.
- 8. Pretreatment of Loop: The loop was cleaned with acetone and alcohol. It was degassed for 18 hr at approximately 204° C under a pressure of $< 2 \times 10^{-5}$ torr until filling with the potassium charge.
- 9. Method of Filling Loop: The primary circuit was subjected to a dynamic vacuum of $< 2 \times 10^{-5}$ torr for 18 hr at approximately 204°C. Potassium was then transferred at 149°C from the dump tank until the proper fluid level in the loop was achieved.
- 10. <u>Startup Procedure</u>: The loop was operated as a natural-circulation loop up to a boiler temperature of 1500°F (816°C). Electrodynamic pump was then used and the system brought up to 1970°F (1077°C).

The pressure in the vacuum chamber was maintained at $< 5 \times 10^{-6}$ torr during the startup period.

11. Test Environment: The present Nb-1% Zr test assembly is operating at a base pressure of 5×10^{-8} torr. The test chamber consists of a Stokes stainless steel vacuum tank connected to two 10-in. diffusion pumps which are backed up by a 4-in. pump.

As an added protection against contamination of the Nb-1% Zr alloy by residual impurities in the vacuum chamber, a direct, skintight tantalum foil wrap was placed on the preheater, boiler, and dryer. Also protected in this manner was the Nb-1% Zr alloy tubing used for pump lines.

12. <u>Instrumentation</u>:

a. Pressure

- 1. In primary circuit Taylor pressure transmitter.
- 2. Vacuum tank Veeco Ion Gage and Varian nude ion gage.
- b. Flow EM-type flowmeter.
- c. Potassium level in condenser resistance-type level device (1 tube)
- d. Temperature measurement Pt₉₀ Rh₁₀/Pt, Chromel-P/Alumel,
 W₅ Re/W₂₆ thermocouples, and multipoint recorders.

13. Description of Components:

- a. Pump Liquid Metals, Inc., electrodynamic pump.
- b. <u>Heaters</u> Tantalum tube resistance heaters designed and fabricated at ORNL (see Section II-C-13).
- c. <u>Boiler</u> 1.9-in.-diam boiler tube surrounded with a 3.25-in.-diam sodium jacket.
- d. Preheater and Dryer 1.9-in.-diam nonjacketed tube.
- e. Condenser A counterflow, NaK-cooled condenser.
- f. <u>Turbine Simulator</u> Three nozzle and blade sets in series in the test section. The first and third nozzles are converging types and the second is a converging-diverging type.
- 14. <u>Sampling Procedure</u>: A potassium sample was collected when the operating charge was placed in the dump tank. A sample will be taken in a similar manner at the completion of each test.



15. <u>Loop Flow Stability</u>: Sudden instabilities in the loop pressure are being encountered at frequencies of approximately one per day. These instabilities result in a maximum boiler temperature fluctuation of 27°C and a system pressure cycle of 50 psia. Completely stable operating periods are presently averaging between 16 and 23 hr.

16. Posttest Procedure:

- a. Complete metallographic examination of all loop components.
- b. Chemical analyses of potassium, NaK, and loop components.
- c. Erosion-corrosion studies on nozzle and blade specimens.
- 17. <u>Major Loop Problems</u>: During the current 1057 hr of loop operation, there has been one shutdown of the EM pump caused by a bearing failure.

The liquid level I-tube indicator in the condenser section is temperature sensitive.

18. Man Power:

- a. Design 2274 man hours.
- b. Operation $-1 \frac{1}{2}$ man per day during operation.
 - (1) engineer 1 man continuously.
 - (2) technician -1/2 man continuously.
- 19. <u>Summary of Results to Date</u>: As of September 16, 1963, the loop has operated 1053 hr at the design conditions listed in Table III-B-1. The test is scheduled to operate for 3000 hr.

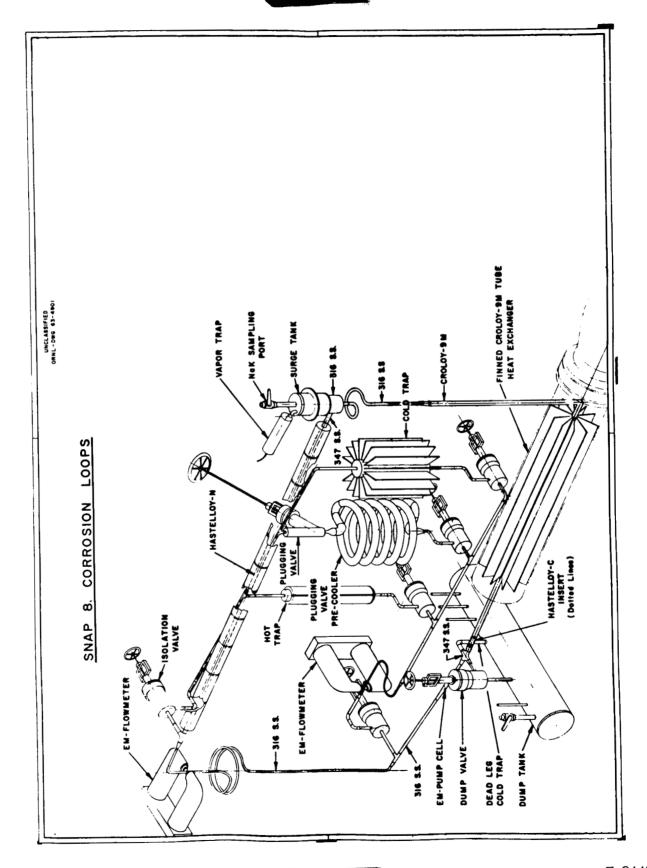




1. <u>Purpose and Sponsor</u>: To investigate the nature of dissimilar metal interaction in the SNAP-8 primary circuit and to study the disposition of hydrogen in this circuit.

Sponsor: NASA.

- 2. Fluids: NaK.
- 3. Forced-Convection Loops:
- 4. Approximate Size: 4 ft × 6 ft (see attached sketch).
- 5. Containment Alloy: Hot leg is 3/4-in.-diam × 0.072-in.-wall Hastelloy N; remainder of loop has 3/8-in.-diam × 0.049-in.-wall type 316 stainless steel tube and l-in.-diam × 0.049-in.-wall 9% Cr-l Mo steel tube. All welded construction.
- 6. Test Conditions: 704°C, 773°C hot leg; 593°C cold leg. Flow rates 720 lb/hr, time 1000-6000 hr.
- 7. <u>Test Specimens</u>: 1-in. tube and 1- × 1/8-in. plates at hottest and coldest locations throughout loop and through the cooler; to be removed at end of test.
- 8. Pretreatment: Degreasing, cold trapping, and hot trapping.
- 9. Method of Filling: Evacuate loop and pressurize NaK.
- 10. Startup Procedure: Not completed.
- 11. Test Environment: Air.
- 12. <u>Instrumentation</u>: Hydrogen analysis Gow Mac Inst. Company, Model 9199, thermal conductivity cell. All others standard.
- 13. Components: EM pump, clamshell heaters, finned-tube heat exchanger, and EM flowmeter.
- 14. <u>Nak Monitor</u>: Plugging valve for oxygen, bucket-type sampler for chemical analysis.



- 15. Flow Stability: Liquid only.
- 16. <u>Posttest Procedure</u>: Metallography, weight changes, electron microprobe analysis.
- 17. Major Loop Problems: Hydrogen injection and hydrogen measurements.
- 18. Man Power: Two engineers, two technicians.
- 19. Results to Date: None.



PRATT & WHITNEY AIRCRAFT COMPANY, CANEL

Middletown, Connecticut

Kenneth Kelly

LCRE Non-Nuclear Systems Test

- 1. To obtain heat transfer data, up to 5 megawatts, for the LCRE primary and secondary coolant circuit and to evaluate system compatibility in long time tests at 5 Mw. AEC Contract AT(30-1)-2789
- 2. Primary Lithium; Secondary 22 Na 78 K
- 3. Forced Convection
- 4. Liquid metal portion could be contained in a cube with a 32×53 ft base and 32 ft high.
- 5. Primary 2.25" OD x .095 wall Cb-1 Zr pipe 1.675 OD x .083 wall Cb-1 Zr pipe

Secondary 3" SCH 80 316 SS pipe 6" SCH 160 316 SS pipe

6. Temperatures Primary 2000 to 935F Secondary 1214 to 685F

Pressure Primary 10 to 35 psig Secondary 10 to 46 psig

Flow Primary 12 lb/sec Secondary 43 lb/sec

Operating Time 2200 on 9-23-63 - test continuing

- 7. The complete system will be examined for corrosion effects.
- 8. Cleaned per CS 309.
- 9. Helium gas pressure
- 10. Test system was evacuated, leak checked and back filled with purified helium. This was repeated until the rate of pressure rise at 400F in the evacuated system indicated minimum off-gassing. System was purged with helium until exit gas was less than 1 ppm H₂O, N₂ and O₂. Cover gas system was accepted as satisfactory when the closed system at 400F indicated a contaminant increase (O₂, N₂ and H₂O) below 0.5 ppm/hour. The system was preheated to 600-1000F, filled with liquid metals and test initiated.
- 11. Static helium at a purity less than 1 ppm for 02, N2 and H20. Monitored by Beckman moisture analyzer and gas chromatograph. Cb-1 Zr alloy section is tantalum wrapped.





- 12. Liquid metal pressure by Moore transmitters.

 Temperature by chromel-alumel thermocouples read out on Minneapolis-Honeywell recorders and indicators.
- 13. I²R heater section, Cb-l Zr alloy (see #5) Shell-and-tube heat exchanger Centrifugal pumps Finned-tube air-cooled heat dump
- 14. Flowing, removable-tube NaK sampler at 3 places at any time.

 Li sampling by vacuum removal from fill tank at start and finish of test.

 Plugging indicator on NaK system during first 3 days of operation until oxygen was too low to measure by plugging indicator.
- 15. Stable within 2%.
- 16. Chemical and metallographic examination
 Dimensional examination
 Photographic record
 General visual examination
 Chemistry of liquid metals for 0, N, C and structural metals
 Examination for leakage
 Special examination of known problem areas which were noted during
 fabrication or operation
 Calibration of instrumentation
 Observation of pipe hanger positions
 Leak checking of inert gas shroud
- 17. Generally most of problems were in support equipment and primarily in the electrical power supply.
- 19. Heat transfer performance tests have been completed up to design requirements of 5 megawatts. All performance conditions and predictions have been met. Endurance test at 5 megawatts is continuing and 2200 hours have been logged up to 9-23-63.





Boiling Potassium Natural Circulation Loops

- 1. To obtain materials compatibility data in boiling, vapor and condensing potassium, AEC Contract AT(30-1)-2789.
- 2. Potassium
- 3. Natural
- 4. Approx. 3 ft x 1 1/2 ft
- 5. l loop Cb-l Zr alloy
 l loop PWC-533 alloy
 Boiler 2.250 OD x 0.125 wall
 Tubing 0.625 OD x 0.062 wall
 Condenser 2.0 OD x 0.125 wall
 Inert chamber TIG weld and anneal
- 6. Boiling potassium at 1040C Cb-1 Zr alloy and 1100C FWC-533 alloy
- 7. Entire loop, plus tensile, tube burst and corrosion specimens in boiling, vapor and condensing regions.
- 8. Cleaned per CS-309
- 9. Vacuum
- 10. Evacuate containment system to 10⁻⁸ Torr and heat loop to test conditions.
- 11. 10⁻⁷ Torr, tubulated and nude ionization gauges Residual gas analyzer
- 12. Chromel-alumel and W-5 Re/W-26 Re thermocouples
- 13. See #5 and #7
- 14. Pretest, after purification with Ti sponge at 1400F for 24 hours, and from fill sample. Planned posttest sample from loop.
- 15. Unknown. Provision made for surface sites in boiler to aid nucleation.
- 16. Chemical and metallurgical analysis of loop and specimens.

 Tensile and tube burst data.
- 17. Unknown
- 19. Initial test scheduled for October 1963.



3/4 Mw Test

- 1. Endurance and compatibility testing of Cb-1 Zr with lithium, AEC Contract AT(30-1)-2789.
- 2. Lithium
- 3. Forced Convection
- 4. Approx. 8' x 3' x 3'
- 5. Cb-1 Zr tubing: approx. 3/16", 1/2" and 1" ID with .100 wall. Inert chamber TIG weld and anneal
- 6. Temperature 2000F/1600F Flow Rate - 495 lb/hr Operating Time -1 loop - 10,000 Hrs.
- 7. Entire loop
- 8. Cleaned per CS 309
- 9. Inert gas pressure fill
- 10. Loop filled after preheat to 1000F
- ll. Continuously purified He. Ta foil wrapped. O_2 and H_2O meters and gas chromatograph. $O_2 < 1$ ppm. See PWAC-381
- 12. Temperature and flow
- 13. I²R heater (AC), EM pump (AC), helium cooled heat exchanger. See FWAC-381
- 14. Pre and posttest lithium sample for oxygen, carbon and nitrogen.
- 15. No problem
- 16. Posttest chemical and metallographic examination of loop. See FWAC-381
- 17. Instrumentation and EM Pump
- 19. No apparent mass transfer attack. See FWAC-381





LCCBK Series

- 1. Compatibility of Cb-1 Zr with lithium, AEC Contract AT(30-1)-2789.
- 2. Lithium
- 3. Forced Convection
- 4. Approx. 4' x 5' x 1'
- 5. Cb-1 Zr tubing: approx. 3/16" and 1/4" ID with .030 wall. Inert chamber TIG weld and anneal
- 6. Temperature 2000F/1000F Flow Rate - 94 lb/hr Operating Time -
 - 3 loops 1000 Hrs. 1 loop - 5000 Hrs.
 - 1 loop 3000 Hrs. on test
- 7. Entire loop
- 8. Cleaned per CS 309
- 9. Inert gas pressure fill
- 10. Loop filled after preheat to 1000F
- 11. Continuously purified He. Two loops using protective coating; three loops using protective Ta foil. 0_2 and H_2O meters and gas chromatograph, $0_2 \le 1$ ppm.
- 12. Temperature and flow
- 13. I²R heater, EM pump (AC), helium cooled heat exchanger
- 14. Pre and posttest lithium sample for oxygen, nitrogen and carbon.
- 15. No problem
- 16. Posttest chemical and metallographic examination of loop material.
- 17. Instrumentation
- 19. No apparent mass transfer attack. See TIM-727

E-2445

Be-NaK-316 SS

- 1. Compatibility of Be and NaK in a type 316 SS system, AEC Contract AT(30-1)-2789.
- 2. NaK-78 (Eutectic)
- 3. Forced Convection
- 4. Approx. 4' x 3' x 2'
- 5. Type 316 SS tubing: approx. 3/8" OD with .050 wall. TIG weld
- 6. Temperature 1000F isothermal and 800F isothermal Operating Time (1000F) 1000 Hrs. (800F) 2000 and 5000 Hrs.

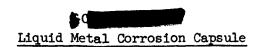
Flow Rate - 850 lb/hr

- 7. Be specimens, some with surface treatment, placed in flowing NaK stream
- 8. Cleaned per CS 309
- 9. Inert gas pressure fill
- 10. Loop filled after preheat to 400F
- ll. Air
- 12. Temperature and flow
- 13. Calrod heat, EM pump (AC)
- 14. Sampling during operation by withdrawal of NaK from flow-through expansion tank for oxygen, beryllium and structural alloys.
- 15. No problem
- 16. Posttest chemical and metallographic examination of beryllium specimens and 316 SS specimen holder.
- 17. --
- 19. Incomplete



LCCDA Series

- 1. Slave system to permit compatibility evaluation of columbium alloy specimens with flowing lithium, AEC Contract AT(30-1)-2789.
- 2. Lithium
- 3. Forced Convection
- 4. Approx. 4' x 5' x 1'
- 5. Cb-l Zr tubing: 1/4" ID with .070 wall + 5/16" ID with 035 and 070 wall Inert chamber: TIG weld and anneal
- 6. Temperature 2000F/1600F
 Flow Rate 175 lb/hr
 Operating Time 1 loop (3) tests 500 Hrs. each
- 7. Cb-l Zr specimens placed in flowing stream
- 8. Cleaned per CS 309
- 9. Inert gas pressure fill
- 10. Loop filled after preheat to 1000F
- 11. Continuously purified He. Loop Ta foil wrapped O2 and H2O meters and gas chromatograph O2 < 1 ppm
- 12. Temperature and flow
- 13. I²R heater, EM pump (AC), helium cooled heat exchanger
- 14. Pre and posttest lithium samples to determine carbon, oxygen and nitrogen
- 15. No problem
- 16. Posttest chemical and metallographic examination of Cb-1 Zr specimens.
- 17. Instrumentation
- 19. Evidence of carbon stability in polythermal dynamic system and of nitrogen transfer from high temperature to lower temperature regions.



- 1. To determine the effects of contaminants on the compatibility of Cb-1 Zr alloy with lithium, AEC Contract AT(30-1)-2789.
- 2. Lithium
- 3. Liquid
- 4. Cb-1 Zr alloy and unalloyed columbium
- 5. Wire
- 6. Specimen .032" x 3 1/4" Capsule - 3/8" x 5"
- 7. Zirconium 0, 0.75 and 0.9 weight percent
 Time (1 min 50 hours)
 Temperature (540-1100C)
 0 50-5000 ppm N 50-1200 ppm grain size
- 8. N 20 ppm, C 300 ppm, O 1000 ppm
 Ti sponge gettered for 4 hours at 870C
 Kjeldahl nitrogen
 Combustion carbon
 (NH3), oxygen
- 9. Filled and welded in a purified argon filled, vacuum glove box
- 10. Sealed capsules and purified argon
- 11. Static
- 12. Not Applicable
- 13. Furnaces
- 14. Minneapolis-Honeywell temperature controllers
- 15. Chemistry, metallography and internal friction analysis
- 16. Metallography and Chemical Analysis
- 17. ± 10%
- 18. Negligible
- 20. a) Lithium will attack Cb and Cb-l Zr alloy when the structural metal contains more than 250 ppm solid solution oxygen.
 - b) Through selective heat treatment, the Cb-1 Zr alloy can contain up to 2 oxygen atoms per zirconium atom plus 250 ppm oxygen in solid solution and not be attacked by lithium.

Ref. FWAC-622 and FWAC-623



AEC Contract AT(30-1)-2789

	bility							-						
	Compatibility	Por	Poor	Good		Good	±		Good	= = =	=		Good	Ξ
	Chemistry	× × ×	×	×		×	×		×	× × :	× ×		×	×
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	Analyses Surface Finish													
	M	××	×	×		×	×		×	××	××		×	×
	Dimen- sional	××	×	×		×	×						×	×
Tests	Type	Static	Static	=		Tilting	E		Static		z		Static	Ε
Capsule Compatibility Tests	Max. Time, Ers.	1,000	10,000	3,000		10,000	5		150	1,000	150		200	=
te Compa	Max. Temp.	2200	1000	=		1000	=		2000	1800	8 =		0011	=
Capsu	Capsule	Cb-1Zr "	316 SS	=		316 88	E		Cb-1Zr		: =		Cb-1Zr	=
	Envir- onment	Argon "	Air	=		Air	=		Argon		: =		Argon	=
	Test Fluid	크" "	Eutectic			Eutectic			Eutectic	Agu : : :	: =		Eutectic	
	Form	Pellets "	Rođ	:		Tab	÷		Cb-1Zr		: :		Rod	E
	Specimens	Uranium Compounds UN UN UN	Boron Carbide Norton Co.	Carborundum Co.	III. Beryllium	Bare	Nitrided	IV. Braze Alloys	T1-22V-15Fe-5N1-8Co	T1-23V-15Fe-8Co T1-23V-20Fe-5N1	T1-23V-25Fe	Beryllium Oxide	Brush Beryl. Co.	BCA
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ABC (Contract AT(30-1)-2789

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y Tests		Type		Static	=		=	=	=	=	Tilting	=	=	=				Static	:		Static	=		Static Tilting	= =
t1b111t	Max.	Time, Hrs.		1,000	=	=	=	=	=	£	2,000	.=		2	3			8	=		%	=		88	8:
Capsule Compatibility Tests	Max.	Temp.		1900			:		=	ŧ	001		=	=				0011	=		2200	=		2400 2200/1800	2200/1800
솅		Capsule Material		Cb-1Zr	÷	=	=	=	=	=	=	=	F	=				Cb-12r	=		Cb-1Zr	=		Cb-1Zr	
		Envir-		Argon	=	=	=	=		=	:	=	Ξ	=				Argon	F		Argon	E		Argon "	2 5
		Test Fluid		3	=	=	=	£	E	=	=	=	=	=				3	=		3	=		크=	= =
		Form		Rod	=	=	E	=	=	=	E	=	=	=				Welded Sheet	:		Welded Sheet	=		Welded Tab Capsule	Tab e
		Specimens	Cermets	T1C-10Mo	T1C-8Mo-2TaC	T1C-8Mo-2WC	T1C-8Mo-2CbC	Tic-5Mo-5Tac	T1C-5MO-5WC	T10-5Mo-5000	K-96'WC-6Co-2(Ta, Cb)0	K-162B TIC-25N1-5Mo-6	WG-8Mo-2000	3735 275 275	Cermet 113505		II & II ALIONS	T1/Cb-1Zr Wel		Ta & Ta Alloys		Ta-10W	Cb Alloys		cb-1zr cb-3zr
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-- Atomic Energy Act of 1954

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